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## Investigation of the electro-optical behaviour of UV-cured polymer/liquid crystal systems

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### Abstract

The electro-optical properties of Polymer Dispersed Liquid Crystal (PDLC) films were studied to understand the relationship between the conditions of preparation, the morphology, and the response to an external electrical field. The PDLC films were obtained by polymerization induced phase separation (PIPS) process using Ultra-violet (UV) cured system composed of a monomeric diacrylate and a nematic liquid crystal using two irradiation doses, 50mJ/cm<sup>2</sup> and 150mJ/cm<sup>2</sup>. The analysis of electro-optical responses were investigated in terms of transmission vs voltage curves as a function of thickness and show similar shapes, low transmission in the off-state and high transmission in the on-state in both cases. The results obtained for 150mJ/cm<sup>2</sup> exhibit slightly lower switching voltages and in particular, the 50mJ/cm<sup>2</sup> samples lead to relatively large dispersed data in terms of threshold voltages.

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### 1. INTRODUCTION

The applications of Polymer Dispersed Liquid Crystal (PDLC) materials are generally based on electro-optical response functions, such as optical shutters, large area displays and smart windows [1-5]. PDLC films consist of micron-sized nematic liquid crystalline (LC) domains dispersed in a polymer matrix. Since the LC molecules can change their orientation under the influence of an applied electrical field, the PDLC film switch under certain conditions between an opaque light scattering off-state to a transparent on-state. In this work, PDLC films were prepared by ultraviolet (UV) irradiation induced polymerization from a homogeneous mixture of tripropyleneglycoldiacrylate (TPGDA) monomer, nematic LC blend E7, and a photoinitiator. When the

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polymerization/cross-linking reactions occur, the LC phase separates from the growing polymer network, forming domains dispersed in the polymer matrix.

The electro-optical performance of PDLC films such as threshold and switching voltages, off-state and on-state transmissions, response times, brightness, and contrast are strongly affected by the conditions of preparation leading to different morphologies in terms of size and shape of the LC domains, and in particular the architecture of the polymer network [6-12].

The authors report on the dependence of the UV-curing conditions on morphology and electro-optical properties by varying both the UV lamp power and the curing time [13–14]. However, the control of the extent of the polymerization/crosslinking reactions is of prime importance for good and reproducible electro-optical performance. It is, therefore, important to achieve complete conversion of the monomer to the polymer network independent of the particular choice of UV light intensities and exposure times. In this paper, the electro-optical response in terms of the transmission as function of applied voltage and film thickness was obtained for the two chosen UV-curing doses ( $50\text{mJ/cm}^2$  and  $150\text{mJ/cm}^2$ ). These results are discussed and compared to observations of the morphology obtained by Scanning Electron Microscopy (SEM) measurements.

## 2. EXPERIMENTAL SECTION

### 2.1. Materials

Tripropyleneglycoldiacrylate (TPGDA) (UCB, Belgium) was used as monomer. The nematic LC E7, an eutectic mixture containing four cyanoparaphenylene derivatives, was employed in this work exhibiting a positive dielectric anisotropy and a nematic-isotropic transition temperature at  $T_{NI}=61^\circ\text{C}$ . 30 weight-percent (wt-%) of TPGDA, 70wt-% of E7, and 2wt-% (of the acrylate mixture) of a photoinitiator (Lucirin TPO, BASF) were mixed together until the blend became homogeneous.

### 2.1. Sample Preparation

For electro-optical investigations, the initial reactive mixture was sandwiched between a glass plate and a  $100\ \mu\text{m}$  thick Polyethyleneterephthalate (PET) sheet (Renker, Germany), both coated with a thin transparent layer of conducting indium/tin oxide. The film thickness was measured by an adapted micrometer calliper (Mitutoyo; diameter 1mm; uncertainty:  $\pm 1\ \mu\text{m}$ ). The precise place of crossing of the laser beam through the film, exhibiting a diameter of about one millimeter, is considered to take repetitive measurements (at least five times) of the thickness of the PDLC sample. Then, measurements of the thicknesses are taken separately on the two supports, namely glass/ITO and PET/ITO, which were cleaned before, once the sandwich (glass-ITO/PDLC film/PET-ITO) was opened. The irregularities of the thickness of the samples were thus taken into account since the measurements were carried out exactly on the crossing point of the laser beam.

### 2.2. Instrumentation

The UV irradiation source used in this study was a Minicure Model MC4-300 (Primarc UV Technology) equipped with a medium pressure mercury arc lamp rated  $80\text{ W/cm}$ . The samples prepared as described above were placed on a conveyor belt; the dose received by the sample was either  $50\text{ mJ/cm}^2$  or  $150\text{ mJ/cm}^2$ .

The electro-optical measurements were carried out by a standard set-up including a laser, a sample holder and a photodiode together with a computer controlled data acquisition for the analysis of the electro-optical properties. The transmission of unpolarized HeNe laser light ( $\lambda=632.8\text{ nm}$ ) passing perpendicular through the PDLC film was measured at room temperature without and upon application of an external electrical field. Several sinusoidal voltage scan up and down cycles with appropriate maximum voltages were applied during a period of 120 s, using a frequency of 145 Hz.

Samples for Scanning Electron Microscope (SEM) studies were elaborated in the same way as described above for electro-optical measurements. After irradiation exposure was finished, the sample cells were opened and immersed in tetrahydrofuran in order to extract the LC. The samples were then coated by a thin platinum layer and

characterized by a SEM (Philips XL-30 field emission gun). This sample preparation technique leads to the appearance of dark holes that were once filled with LC. The magnification used in our experiments was about 20000x using an acceleration voltage of 10kV.

### 3. RESULTS AND DISCUSSION

Important physical properties of PDLC films such as the electro-optical response and the mechanical behavior are directly related with the conditions under which the materials are obtained. In particular, the kinetics of photopolymerization and the phase separation behavior of the monomer/LC blend govern the architecture of the obtained polymer network. In order to analyse the extent of formation of the polymer network, the relationship between the conversion of the monomer and the curing conditions was investigated. It is evident that high monomer conversions should be reached to minimize undesired effects of unreacted monomers or dangling chains. FTIR spectroscopy was applied as a versatile method to investigate the extent of curing via carbon-carbon double bond conversion. A difunctional acrylic monomer was used in this work giving rise to a chemically crosslinked polymer network. The results obtained from FTIR measurements indicate a complete monomer conversion for samples cured with  $50\text{mJ}/\text{cm}^2$  and  $150\text{mJ}/\text{cm}^2$ , by considering the peak height of the absorption band at  $1637\text{cm}^{-1}$ .

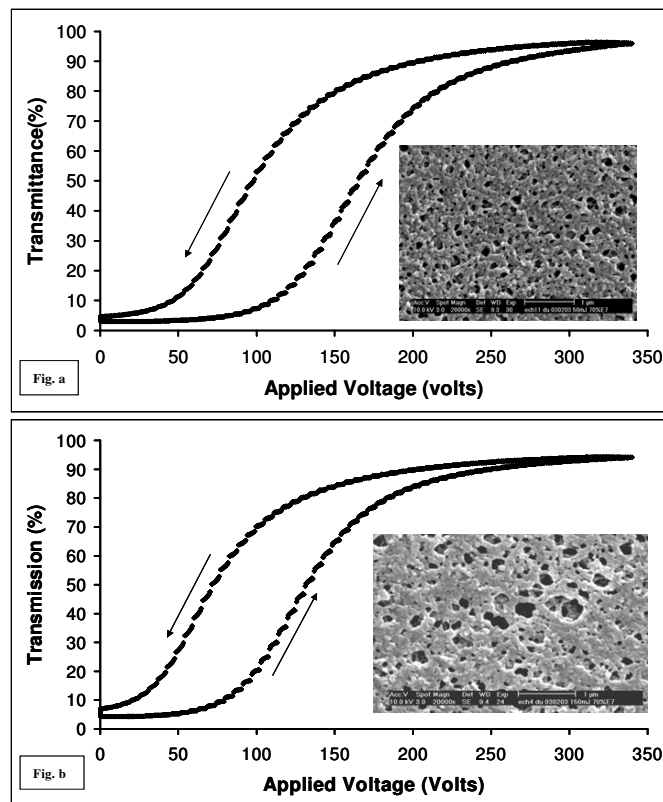


Fig. 1. Electro-optical response of  $25\ \mu\text{m}$  thick PDLC films a)  $50\text{mJ}/\text{cm}^2$ , b)  $150\text{mJ}/\text{cm}^2$ . The inset exhibit the corresponding sample morphology obtained by SEM.

Figures 1a and 1b exhibit transmission vs. voltage curves of 25 $\mu\text{m}$  thick PDLC films cured with 50mJ/cm<sup>2</sup> and 150mJ/cm<sup>2</sup>, respectively. Both electro-optical response curves show low transmission values in the off-state, high transmission in the on-state, and similar shapes characterized by a slow increase of the transmission upon application of an electrical field. Furthermore, the samples show a relatively large hysteresis i.e. in an increasing field, the transmission at a specific voltage is lower than the transmission at the same voltage in a decreasing electrical field. The electrical field necessary to switch the 50mJ/cm<sup>2</sup> film from an opaque to a transparent state is slightly higher than for the 150mJ/cm<sup>2</sup> sample as can be seen by comparing the electro-optical response curves shown in Figures 1a and 1b.

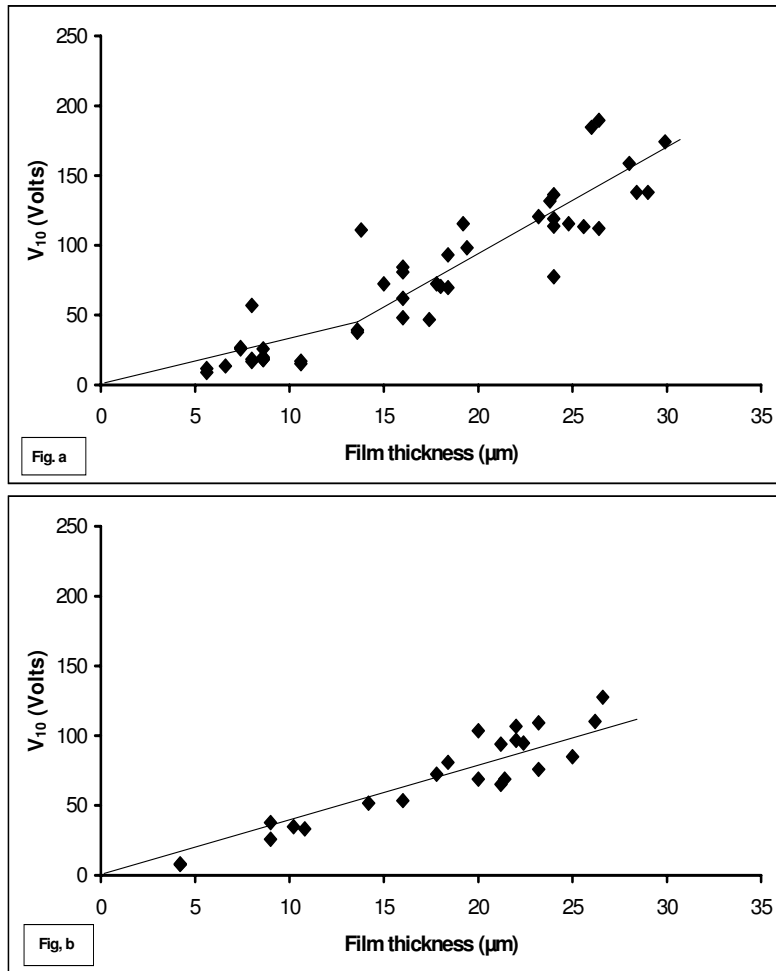


Fig 2. Threshold ( $V_{10}$ ) voltages as a function of film thickness for UV-cured samples, a) 50 mJ/cm<sup>2</sup>, b) 150 mJ/cm<sup>2</sup>.

The electro-optical properties can be explained by the analysis of the corresponding sample morphologies which are displayed as insets in these figures. Large size distributions of irregularly shaped domains were observed in both cases and are in good agreement with the slow response to the applied electrical field: LC molecules in larger domains orient already at low electrical fields whereas LC molecules in smaller domains need higher voltages to

orient due to the higher curvature of these domains. The morphology analysis reveals increased domain sizes of the 150 mJ/cm<sup>2</sup> sample compared to 50 mJ/cm<sup>2</sup> which lead to the observed shift of the transmission vs. voltage curve to lower values.

Figure 2 shows the threshold voltages as a function of film thickness for a) 50 mJ/cm<sup>2</sup> and b) 150 mJ/cm<sup>2</sup> samples. Widely dispersed results were found in the case of the lower UV-dose and the experimental data cannot be described by a linear relationship. However, the results can be fitted by a combination of two linear regression curves with a change of the slope around a film thickness of 14 μm. On the other hand, the results obtained for the 150 mJ/cm<sup>2</sup> sample can be described by a single linear fit. Also the performance of these films in terms of electro-optical responses is to achieve a high contrast ratio  $T_{on}/T_{off}$ . The intensity of transmitted light at right angle was recorded as a function of time starting at the time where the applied electric field is removed; and data are taken at room temperature for two doses.

Another important parameter which was investigated in this work is the time relaxation of these PDLC systems after applying an electrical field to reach the OFF state of both 50 and 150mJ/cm<sup>2</sup> cured films. Figure 3 shows two distinct relaxation processes, a slow and a fast processes. The curves are plotted in the descending order of UV doses curing, meaning that the relaxation is faster as the dose is increased.

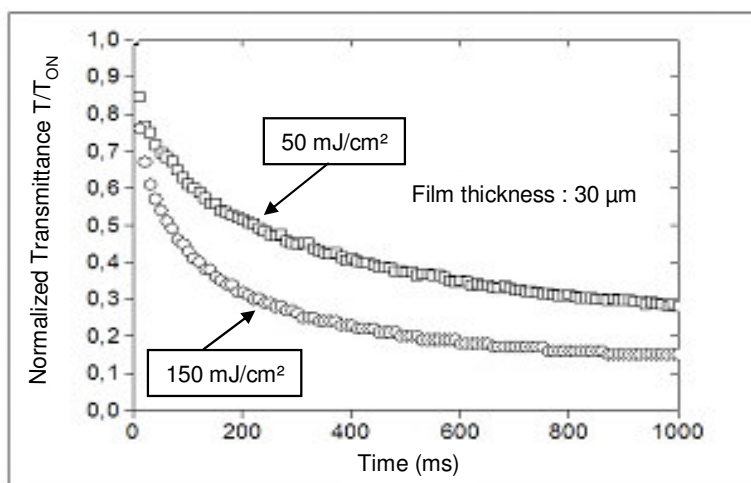


Fig. 3.  $T/T_{on}$  versus time for UV-cured PDLC films at 50 mJ/cm<sup>2</sup> and 150 mJ/cm<sup>2</sup>. The electric field applied corresponds to a rectangular pulse lasting 60s at the voltage applied and the origin corresponds to the time where the field is removed.

#### 4. Conclusion

PDLC films prepared by UV-irradiation using a PIPS process were studied in order to understand the effect of UV-dose on electro-optical performances of these composite materials. The results obtained from morphologies and some electro-optical properties of these samples were different comparing the two doses. The transmission vs voltage curves show similar shapes which can be explained by a large droplet size distribution observed in both cases as shown by morphology analysis obtained by SEM. The threshold and saturation voltages are slightly higher for the lower UV-dose samples. This behavior was expected because the droplet sizes are smaller in comparison with those corresponding to higher doses. These results are in good agreement with the obtained film morphologies showing relatively small domain sizes in the case of the lower dose. The dependence of the threshold voltage on the film thickness was found to be different for the two cases. The results obtained from the higher UV dose indicate, as expected, a linear variation of the threshold voltage as a function of the film thickness and for the lower dose the results are widely dispersed and cannot be fitted by a single linear slope. The relaxation is faster as the dose is

150mJ/cm<sup>2</sup>, a possible explanation of this phenomenon could be probably the morphology of these films as seen above and the architecture of the polymer network that can be formed differently for the two irradiation doses.

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